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SPACE SIMULATION ULTIMATE PRESSURE LOWERED TWO DECADES BY REMOVAL OF DIFFUSION PUMP OIL CONTAMINANTS DURING OPERATION

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ABSTRACT

The complex problem why large space simulation chambers do not realize the true ultimate vacuum was investigated. Some contaminating factors affecting diffusion pump performance have been identified and some advances in vacuum/distillation/fractionation technology have been achieved which resulted in a two decade or more lower ultimate pressure. Data are presented to show the overall or individual contaminating effect of commonly used phthalate ester plasticizers of 390 to 530 molecular weight (M.W.) on diffusion pump performance. Methods for removing contaminants from diffusion pump silicone oil during operation and reclaiming contaminated oil by high vacuum molecular distillation are described.

SUMMARY

Identification of contamination problems associated with space simulation test programs, their overall or individual effect on diffusion pump performance and its rapid deterioration on silicone diffusion pump fluids were studied. Diffusion pump performance as measured in terms of ultimate vacuum, shall be compared between identically contaminated oils in diffusion pumps with or without self-purification capabilities.

A series of experimental scale model glass diffusion pump vacuum systems evolved which investigated certain physical properties associated with evaporative surface behavior phenomena versus ultimate realizable vacuum. This research indicates that contaminants divide into light-end and heavy-end constituents inside the diffusion pump. Boiler fluid separation-purification design innovations successfully remove the heavy-end fractions from the evaporative surface by skimming-bleed methods and light-end fractions by vacuum distilling/bleeding action at temperatures of approximately 130° C in the foreline. These diffusion pump innovations, which are discussed and illustrated, resulted in a two decade or more lower ultimate pressure.

Documentation reveals the progressive improvement in molecular

release rate corresponding with the increase in diffusion pump performance and resultant lower ultimate pressure. This documentation consists of infrared thermovision polaroid pictures, slow and high speed movies, and 35mm slide photographs of the diffusion pump boiler evaporative surface and jet assembly molecular flow patterns.

Other significant results revealed were:

a. Contamination in the pump fluid limit the efficiency of diffusion pumps by 100 to 1000 times as indicated in terms of ultimate vacuum obtained.

b. Contaminants, commonly found in large vacuum chambers, that adversely affect silicone oil diffusion pump performance were identified as phthalate ester plasticizers. These plasticizers are utilized in cable insulation jackets, alkyd paints, electronic components, etc.

c. Contaminated (silicone) diffusion pump fluid can be successfully reprocessed by a triple cut (two light-end and one heavy-end) high vacuum centrifugal molecular distillation method. Reclaiming process nets an approximate 75±5% yield of oil capable of achieving an ultimate vacuum close to that of new DC-705 oil.

d. The effect of specific or mixtures of various phthalate plasticizer contaminants on performance of diffusion pumps were measured. Of all the plasticizers studied di-isooctyl phthalate (DIOP) (390 molecular weight, M.W.) had the most deteriorating effect on DC-705. Di-isodecyl phthalate (DIDP) (446 M.W.) di-octyl phthalate/di-2-ethlhexyl phthalate (DOP/DEHP) (390 M.W.), and di-tridecyl phthalate (DTDP) (530 M.W.) all achieved nearly the same ultimate vacuum. Higher molecular weight contaminants (such as 530 M.W. for DTPP) in DC-705 does not guarantee a lower ultimate pressure.

INTRODUCTION

Large space simulation chambers are not presently capable of realizing their true ultimate vacuum because of contamination limiting the efficiency of their diffusion pumps. Present diffusion pumps are not totally suitable for self-purification; that is capable of removing or preventing contamination of the pump fluid. Since a rapid degradation in pump performance occurs when exposed to sources of outgassing contaminants associated with space simulation test programs, a study of the fundamental performance limiting causes was initiated with the following objectives:

a. Identify contaminants commonly found in large vacuum chamber that adversely affect silicone oil diffusion pump performance.

b. Measure the effect of various contaminants on performance of diffusion pumps.

c. Develop means of removing contaminants from oil by vacuum distillation during diffusion pump operation.

BACKGROUND THEORY AND DEVELOPMENT

Overall Problem Background--While performing initial Brayon Power System testing in the 800 000 cu ft (22.7×10⁶ liter) SPF test chamber, accumulations of condensable volatiles were observed on the 100 ft (30.5 m) diameter floor in 1969. NASA, Kennedy Space Center (KSC) Analytical Laboratory analyzed these condensables by a combination of column and gas chromatography, infrared, emission and mass spectroscopy. Samples were found to consist primarily of phthalate esters, an aromatic amine plus smaller amounts of a hydrocarbon oil and silicone oil.

During 1970 and 1971 while conducting Skylab Payload Shroud Jettison tests at a simulated altitude of 300 000 ft (3.3×10^{-4} Torr) the DC-705 diffusion pump oil became contaminated due to the large outgassing load, see Figure 1. A complete oil change costing $30 000 was implemented. The magnitude of the diffusion pump oil contamination problem was revealed by the 1.5 decade loss in performance of new DC-705 after twenty-five operational hours of Skylab Shroud testing.

In order to thoroughly understand the outgassing contamination problem, associated with large space simulation test programs, a materials inventory search was undertaken to establish which compounds of those presently being used were the most volatile. Thirty-nine major materials were used in quantities ranging from 1580 kg (3500 lb) to 4.54 kg (10 lb) in conjunction with the Brayton and Skylab test programs. Those materials that are formulated with plasticizers, found in the analyses of eight DC-705 oil samples, shall be listed and described.

Background Information Relative to Contamination of Vapor Pumps--
Diffusion pumps commonly called inverted condensation pumps or vapor pumps are dynamic equilibrium systems. These pumps are dependent on the respective vapor pressure-molecular weight-temperature interrelationships associated with a mixture of working fluid constituents. As such, diffusion pumps are stills, a commonly known fact.

For years many have been recommending that clean pump fluids generate the best ultimate vacuums. [6-10] Huntress does state that less than one part per million of certain types of volatile contaminants was shown to alter seriously the effective vapor pressure of the working fluid. Removing contaminants though, has not been easy. Cryo trapping them on baffles is not a complete corrective measure since eventually saturation occurs causing vacuum system pressure spikes and contamination of the test article. Also upon baffle warmup, condensed contaminants may drip back into the pump fluid. Since occasional diffusion pump boiler erruptions, sometimes called burps, have been considered undesirable from a backstreaming standpoint, it has been the practice to minimize or eliminate vigorous boiling evaporative surfaces by design. [10]

Other contamination related factors are: large outgassing test hardware in large space simulation chambers which are difficult and expensive to clean; diffusion pump oil is too costly to be periodically replaced or repeatedly reprocessed when quickly contaminated; and elaborate baffle systems too expensive to purchase and/or operate. A logical course of action was to investigate methods to increase the self-cleaning, self-purification-separation capabilities of our present diffusion pumps (stills).

Background Distillation Information--The art of distillation was practiced before the Christian era by the Egyptian temple priests. However, molecular distillation is relatively new. In the early 1950's, Hickman found while working with pot stills that when liquids are permitted to evaporate rapidly without restriction into a high vacuum environment their surfaces often separate into two zones of widely different appearance and properties. [6,7] One, which is in rapid self-motion called "WORKING" and the other, which is acquiescent and photographically highly reflective called "TORPID." An evaporative surface exhibiting a dual working/torpid behavior pattern or patterns was called "SCHIZOID." Torpidity, a floatation phenomena, is believed to be due to traces of impurities that block the surface during rapid evaporation.

Development Approach--The self-cleaning 1×10^{-6} SPF scale model glass vacuum system was designed to meet the following objectives:
    a. Understand the fundamental phenomena of torpidity and its effect on achieving ultimate vacuum or maximum pump speed.
    b. Determine methods and operating parameters for preventing torpidity or removing a torpid evaporative surface condition in order to generate a 100% working surface.
    c. Determine percent of contaminants inducing torpidity and/or its related ultimate vacuum limiting effects.
    d. Determine methods and operating parameters for removing light-end contaminants in the forearm.
    e. Determine methods and operating parameters for removing heavy-end contaminants from the boiler fluid.

Supporting Keys to Self-Cleansing Design--
    a. The concepts of torpid/schizoid/working evaporative surfaces. Torpidity as a surface phenomena subject to removal by surface skimming resulting in a schizoid evaporative surface.
    b. Existance of multiple groups of contaminant by spectroscopy analyses. Heavy-end contaminants grouping at approximately 700 M.W., and light-ends peaking at approximately 300 M.W. established the necessity for a triple cut molecular distillation process. Nearly two to three

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times the quantity of light-ends exist than the quantity that exists of heavy-ends.

c. The final key was within normal vacuum fractionation distillation ranges.[11,12] Hickman's early condensation pump designs, all use the alembric conept of liquid-vapor equilibrium in the vertical stage. Acute observation revealed that when the alembrics fill up, the heavier volatile constituents overflow back into the pump boiler and are never fully discharged.

APPARATUS AND PROCEDURE DISCUSSION

Analysis Apparatus--The following equipment was utilizied to study the material outgassing contamination problem:

    a. Computerized digilab FTS-20 infrared-fourier transform spectrometer (at KSC)
    b. RMU GL Hitachi mass spectrometer and 900 Perkin-Elmer gas chromatograph linked with a 6201 Varian data system (at GSFC)
    c. Q-250 residual gas analyzer (RGA) (at LeRC)
    d. Individual 502 Water Associates liquid chromatographs (at KSC and GSFC)
    e. Gas chromatography mass spectroscopy units (at KSC, GSFC, and LeRC)
    f. AGA thermovision (at LeRC)
    g. Quartz crystal microbalance (at GSFC and LeRC)
    i. Environment control-instrumentation systems (at LeRC)
    j. SPF - 1×10^{-6} scale model glass vacuum systems (at LeRC): G-4, modified G-4, 1st generation self-cleansing and 2nd generation self-cleansing

Standard Diffusion Pump Oil Performance Test Setup-- A single stage glass diffusion pump (designated a G-4) having a rated pump speed of 4 liters/sec was used for all oil performance comparison tests, see Figure 3. The G-4 vapor pump has long been used by the pump fluid and vacuum system manufacturers for comparing performance of various pump fluids. The prime reasons for using glass were the control over material contaminating factors plus the benefit of visually observing the operational phases of the vapor pump.

Molecular Distillation, Silicone Oil, Reprocessing Setup--Conventional methods for reclaiming contaminated pump oil are relatively ineffective or expensive. A new method was developed for reclaiming contaminated vacuum diffusion pump oil by high vacuum molecular distillation processes.[3] A high vacuum centrifugal molecular still was utilized to centrifugally separate volatile contaminant/component compounds from the oil at their individual respective evaporation temperatures. Preheated dis-

tilland feedstock is metered onto a heated rotating evaporator disk where separation occurs. The light end contaminant vapors are condensed on the cool inner surface of the bell jar. The pure oil and heavy end compounds are not vaporized and are spun off the rotating disk by centrifugal force, caught in a water-cooled gutter and collected as a residue. Final cut distillation is performed in a similar manner except that the pure oil is vaporized, condensed, and recovered as a distillate with the heavy end contaminants being separated as a residue. See Figure 2 for reprocessing setup.

Acceptance criteria of molecular distilled-reprocessed DC-705 is attainment of a 5×10^{-6} Torr ultimate vacuum within 4 hr using the G-4 pump oil performance setup. Failure to meet the acceptance criteria indicates the residual existance of additional light-end contaminants, which necessitates another light-end cut distillation. A high vacuum molecular wiped-film type still may also be used successfully. For additional theory relative to molecular distillation see Burrows[4] and Watt[5].

More extensive information on the reprocessing set up, including equipment description, molecular still operating philosophy, distillation processing and G-4 solvent cleaning preparations plus operation procedures may be obtained from the Technology Utilization Officer at NASA/LeRC reguarding Reference 3.

Second Generation Self-Cleansing 1×10^{-6} SPF-Scale Model Glass Vacuum System--A three jet glass diffusion pump, having approximately the same size boiler and resultant evaporative surface as the (G-4) single stage diffusion pump, was constructed to study the effects of inserting phthalate plasticizers and other contaminants directly into the pump fluid or into the chamber during pump operation (see Fig. 4(a)). The necessary removal of and identification of these volatile contaminating constituents are required in order to prevent the rapid deterioration of the pump fluid and the resultant reduced pump efficiency. See Figure 4(b) for self-cleansing (purification-separation) design innovation details.

Since the second generation self-cleansing 1×10^{-6} scale model apparatus generates ultra pure pump fluid with resultant lower ultimate chamber pressures, these innovations shall be described. Follow-on foreline vacuum distillation designs have been fabricated which should further improve the separation capability of contaminants in pump fluid, see Figure 4(c). Because these designs have not generated sufficient performance data to allow comparison as of this writing no further discussion will be included.

The design solution unfolded as a merger of the three interrelationships: (1) surface skimming heavy-ends, (2) periodically draining different light-end fractions until the foreline temperature when heated externally stays stabilized at a maximum of about 130° C, and (3) obtain-

ment of a 100% working evaporative surface indicated by a 5° to 7° C boiler vapor-liquid temperature difference. Optimum performance was achieved with a boiler fluid level being 5mm±1mm in depth. Any one of the three concepts by itself or paired with another will not allow achievement of the optimum pump efficiency condition possible with a continuous 100% working evaporative surface. Analyses showed the pump boiler fluid improves to a purity of 99.8% or better when the evaporative surface exhibits a continuous 100% working condition. The critical point in the ratio of contaminant to pure liquid versus lowest realizable ultimate pressure is still uncertain. This is primarily due to the existing noise levels in the spectroscopy analysis system.

Foreline Purification--In the forearms of Figure 4(b) and (c), a distillation process occurs in which mixtures of soluble liquids, carried along by the pumps throughput, are separated by evaporation and condensation, whereby the condensed part becomes richer in the most volatile constituents. This condensate, high in percentage of contaminant, is periodically drained into a vessel of slightly lower pressure while the diffusion pump is operating. If this high volatile condensate, or reflux is allowed to return to the diffusion pump boiler the volatile constituents are vaporized, move up the Christmas tree and are discharged out the respective jets to be condensed on the cooler wall of the diffusion pumps barrel. Both pure fluid and contaminated fluid molecules follow similar paths. The primary distinction between the two types of vapor or condensates being the dwell time each exists on the diffusion pump barrel wall surface. More volatile molecules for a specific temperature and pressure tend to break the polar and dispersion bond forces and thus become free to "backstream" (also known as, "reverse fractionation") back up into the chamber. Other less volatile constituents are carried as reflux up into the forearm of the diffusion pump where reverse fractionation again occurs.

Surface Skimming--Means were built into the self-cleansing pump to remove impurities from the evaporative surface. (See Figs. 4(a) and (b)). DC-705 silicone diffusion pump oil is a discrete phenylmethyl siloxane species and is chemically a pentaphenyltrimethyltrisiloxane having a molecular weight of 546. Thus being basically a pentamer, by hypothesis, when the oil becomes contaminated and cracks it may become a trimer and/or a heptamer. This was essentially proven by analyses, identification of 700 M.W. group peaks, and the heavy-end molecular distillation cut. Skimming the evaporative surface, analyzing the sample and discovering small quantities of phthalate esters proves the validity of the hypothesis.

RESULTS AND DISCUSSION

Contaminating Compounds Affecting DC-705

An inventory search of 99% of the SPF materials utilized for the Brayton and Skylab Space Simulation Test Programs revealed thirty-nine different components. Only those materials that were found to be volatile compounds (extensive outgassing materials) are included with their respective analyses in Table I(a).

Standardized outgassing tests, to determine the percent weight-lost and percent of volatile condensable materials (VCM), was performed by exposing a material sample to a 1×10^{-6} Torr pressure at 125° C for 24 hr or more.[13] Making up the percent of weight losses are noncondensables (true gases), and hot vaporous condensables (VCM) which are deposited on a 23° C surface. Differential thermal analysis (DTA) performed at Kennedy Space Center (KSC) subjected a similar sample to progressively higher temperatures at a 10^{-4} Torr environment while measuring the decreasing weight. Comparing outgassing and DTA test results for typical volatile cable insulations sample 5(a) and 6(a), 5(b) and 6(b) revealed a close correlation between the two methods. Per DTA, volatile materials that were subjected to 3000 hr of a 10^{-5} to 10^{-4} Torr vacuum had nearly the same weight loss (90 to 97%) as did those samples that were never exposed to vacuum.

Comparing alkyd paint samples 2(c) and 4(b) and 2(a) and 3(a) outgassing weight loss or percent VCM data revealed a greater difference in outgassing rates, 55 to 150% for nearly 3000 vacuum hours difference. Nearly the same outgassing rate of 88% existed for the relative short time of 600 hr difference in vacuum exposure. Per Reference 13, one percent total outgassing and 0.1 percent volatile condensables should be the outer limits on material acceptable for spacecraft use, especially for long-term missions.

Identification of Worst Outgassing Materials and Compounds

As previously specified, all materials listed in Table I(a) were found to contain volatile compounds and as a result would be unsuitable for deep-space missions by violating the foregoing outgassing acceptance criteria. However practical considerations in operating large space simulation chambers and conducting large test programs is always a compromise between schedule and availability of materials. Such was the case with Belden's #8404 two conductor or four conductor shielded cable. The manufacturer states it is 25% plasticized with di-isodecyl phthalate (DIDP) 446 M.W.. Test results indicate it was the worst offender in the chamber, followed by polyvinylchloride (PVC), neoprene, alkyd oil paints, etc. Independent KSC/GSFC chemical analyses and

References 14 to 16 reveal the existance of phthalate esters, carbonaceous materials, aromatic amine, antiozdant, organic salt, and to a lesser degree nonphthalate type ester resin in the material samples listed in Table I(a).

The foregoing contaminants were also detected in analyses of various DC-705 oil samples listed in Table I(b) and (c).

Effect of Various Contaminants on Performance of Diffusion Pumps and DC-705 Silicone Oil

Various Aged Oil Performance Comparisons--The G-4 single stage glass diffusion pump system illustrated in Figure 3 was primarily used as an oil performance-calibration-evaluation-device. The rapid deterioration of silicone pump fluid in an environment that is filled with (high outgassing) materials is illustrated by Figure 5. New DC-705 in a typical G-4, 4 hr test consistantly achieves an ultimate vacuum of 1×10^{-6} Torr. New DC-705 after being exposed to a high outgassing environment for 25 hr in conjunction with performing Skylab Payload Shroud Jettison Test No. 3, achieved only 4.8×10^{-5} Torr for a resultant performance loss of nearly fifty times. Significant was the 0.3 decade performance gain of 339 hr old oil to approximately 2×10^{-5} Torr. Interest in the 0.1 decade pressure fluctuation at the 4 hr point of the standard G-4 performance test lead to confirming the repeatibility and/or the extent of such fluctuations in pressure. Since any changes in ultimate pressure are indications of changes in pump efficiency, which is directly correlatible to pump speed, the approach of improving the self-cleansing capability of the diffusion pump as a still was embarked upon.

After 943.5 hr of operation another oil sample was drawn from a 48-in. pump and its performance checked. This 943.5 hr old oil for all practical purposes was identical to 3000 hr oil in performance. However, color was slightly lighter for 943 hr oil. As previously described in the foregoing theory discussion, the G-4 now was operating at a higher equilibrium pressure, limited by the reverse fractionation rate of the contaminated pump oil which in effect is dependant on the total number of contaminants, their respective quantities, and their respective vapor temperatures and pressures.

Contaminant Effect on Oil Performance--Study of individual plasticizers, their effect when mixed into new DC-705 at various percentages and the resultant effect on ultimate vacuum are revealed in Figure 6. Three milliliters (ml) of di-isooctyl phthalate (DIOP) into 60 ml of new DC-705 for a total of 4.78% contaminant achieved an ultimate vacuum of 2.2×10^{-5} Torr. However, 5 ml of DIOP into 60 ml DC-705, 7.7% total contaminant mixture achieved only a 2.3×10^{-4} Torr ultimate vacuum representing a 100 times loss in efficiency. The 0.1 decade fluctuation was ob-

served to be associated this time with the filling of the top alembrics in the vertical portion of the G-4 glass diffusion pump, and the following reduction in ultimate vacuum pressure rise corresponded to the spill over of contaminant concentrated fluid from these top alembrics. Thus the logical solution was to remove the fluid from these top alembrics periodically which was accomplished in a modified G-4 diffusion pump. The other phthalate plasticizer contaminant runs with new DC-705 followed the general increase in pressure trend with corresponding increase in percentages of DIOP contaminant. However, in this paper only the data associated with 7.7% of the following contaminant mixtures were included for di-isodecyl phthalate (DIDP), di-octyl phthalate/di-2-ethylhexyl phthalate (DOP/DEHP), and di-tridecyl phthalate (DTDP). Suprising was the slightly higher ultimate pressure obtained with DTDP (M.W. 530) contrary to the expectation that higher molecular weight contaminant would affect the DC-705 mixture the least. Evidently additional study is required in this area.

Reclaiming Contaminated Vacuum Diffusion Pump Oil

Molecular Distillation Reprocessing Results--The triple cut distillation process, utilizing either a high vacuum centrifugal molecular still or a high vacuum molecular wiped-film type still, successfully reclaimed 3000 hr old DC-705 oil.[3] Contaminated oil that was once considered worthless was recovered yielding 71.5% (335 kg reclaimed from 469 kg) resulting in a $34 700 annual savings. The 4 hr G-4 acceptance performance test achieved 2×10^{-4} Torr for contaminated 3000 hr oil versus 1.65×10^{-6} Torr for triple cut reprocessed oil.

Molecular Distillation Reprocessing Other Silicone Fluids--The molecular still operating philosophy and distillation process/procedures and parameters would be similar for contaminated DC-704 tetramethyltetraphenyltrisiloxane. However, distillation temperatures would be slightly reduced because of its lower molecular weight of 484.

Philosophy of Reprocessing by Molecular Distillation--DC-705 oil is chemically a pentaphenyltrimethytrisiloxane having a molecular weight 546. As the oil becomes contaminated and/or cracks, an unknown amount may be a trimer and/or heptamer. Volatile constituents are separated from the pure DC-705 oil by precut distillation where light-end contaminants such as phthalate esters (common cable insulation plasticizers) ranging between 100 and 400 M.W. are removed as a distillate and heavy-end contaminants with molecular weight about 700 being removed as a residue during final cut distillation.

Economic Limitations of Molecular Distillation Reprocessing--Using high vacuum centrifugal molecular distillation as an occassional method

of reclaiming a high priced fluid such as DC-705 or DC-704 is certainly economical. A CMS-15A single molecular still reprocessed the contaminated SPF oil in about one week at a continuous 4 gal/hr rate per each distillation cut. However, if the diffusion pump oil deteriorates as rapidly as experienced at SPF, (see Fig. 5) the necessity of repeated oil changes and reprocessing becomes impractical because of the hinderance to the test program. A solution to this problem would be a totally self-cleansing diffusion pump.

Second Generation Self-Cleansing 1×10^{-6} SPF-Scale Model Glass Vacuum System

Figures 7(a) to (c) are the product of one continuous test lasting 40 days with the 1×10^{-6} SPF-scale model self-cleansing vacuum system. Figure 7(d) data was obtained with the same test apparatus. The same 65 cm (2.5 in.) diam by 8mm thick sodium chloride (NaCL) single crystal specially polished was used for all comparison tests shown in Figure 7. Because the discussion of critical interrelated parameters are time correlated and effected by specific events, letters and/or numerals are utilized designating significant data. Numbers assigned to top-chamber pressure or boiler fluid depth parameters designate a specific serialized photographic condition, for typical corresponding slide, movie frame, thermovision monitor movie frame and polaroid picture of a torpid/schizoid/working surface (see Fig. 8).

Figure 7(a) Phase I, New DC-705 Oil Performance Test Data, Increased Purification of New DC-705 Nets 1.5 Decade--The second generation
vacuum system incorporating contamination removal technology was initially charged with 55 ml of new DC-705 oil (see Fig. 4(a) and (b)). After three days of operation the upper ion gage in the 1×10^{-6} scale chamber was indicating a consistant 5×10^{-5} Torr pressure. Point "A" on the fourth day reveals the effect of putting on all ion gages and slowly increasing the foreline heater up to full rated capacity (35 W). A 100% working surface as previously defined existed 10% of the time. The effect of removing approximately 6 ml of light-end (L.E.) fractions in the foreline, 2 ml (L.E.) from each drain line, revealed a 60° F increase in foreline temperature almost instantly, along with a correspondingly 0.6 decade lower chamber pressure. Also an increasing trend in boiler vapor and liquid temperature was observed. After a night in this configuration the variation in boiler vapor and liquid temperatures were diminishing. The 100% working surface periods were now increased in duration and in frequency to 25% of the time.

On the fifth day, the initial effect of removing approximately 7 ml of heavy-end (H.E.) fractions from the boiler evaporative surface, was to increase the 100% working surface periods to 50% of the time and to sta-

bilize the boiler vapor and liquid temperatures. Only a slight reduction in chamber pressure resulted.

On the sixth day, three additional millileters of L.E. were removed from the top alembric of the foreline. As experienced on the fourth day, foreline temperature increased to 275° F, boiler vapor rose to 435° F, while boiler liquid temperature reached 455° F, and the jet molecular release rate was visually observed to intensify resulting in a decreasing trend in ultimate chamber pressure. The 400 W heater input to the boiler was reduced at point "B."

Data points "C," "D" and "E" are associated with establishing the optimum boiler heater input for best diffusion pump performance corresponding to the lowest chamber pressure. Chamber pressure now ranged between 7×10^{-7} and 2×10^{-6} Torr from the 10th day to the 12th day while evaporative surface was working 75% of the time.

On the 12th day, with the boiler's evaporative surface behavior described as 100% working 75% of the time, foreline temperature stabilized at about 250° F, boiler vapor temperature was stable within 405° to 420° F, and boiler liquid temperature varied from 430° to 440° F. The only limitation remaining was the achievement of a continuous 100% working evaporative surface. Thus addition 4 ml of L.E. fractions were removed in 2 ml steps within an hour from the top alembric in the foreline. This reduced the boiler fluid depth to approximately 4mm when this alembric again became full within 20 to 22 min. These removal of minute quantities of unknown L.E. fractions achieved a continuous 100% working evaporative surface behavior pattern for the first time. Further removal of 2 ml of L.E. from the top alembric resulted in a decrease in ultimate chamber vacuum (See movie take #31). Foreline temperature reduced by 25° to 250° F, however, fluctuations in boiler vapor and liquid temperature were for all practical purposes nonexistant. Both parameters were near their all time weighted average maximum point, respectively 425° and 442° F. The boiler fluid depth was now about 3mm for 75% of the evaporative surface area, but due to vapor recoil action, 20 to 30% of the boiler bottom was bare, except for what appeared as a minute molecular film layer. This layer was surmised to be left or caused by the ever moving-vigorous boiling evaporative surface. See photograph No. 21 for an equivalent visual illustration. Point "F" indicates where 10 ml of new DC-705 was directly added to the diffusion pump boiler at a slow insertion rate of 1 ml/min causing a small rise in chamber pressure. The foregoing addition of new DC-705 into the boiler brought the fluid level back up to approximately 7mm. However since the chamber pressure rose approximately 0.5 decade to 5×10^{-6} Torr, and the boiler evaporative surface was again 100% working 75% of the time, the remaining L.E. and H.E. contaminants in new DC-705 was suspect. Upon removing 2 ml of L.E. and approximately 0.5 ml of H.E. chamber pressure decreased to between 1×10^{-6} to 2.5×10^{-6} Torr.

Since it is very difficult to measure a highly dynamic liquid depth and/or determine the exact amount being removed through relatively long glass tubulation, the accuracy on all fluid depth and volumn measurements is ±1mm/±1 ml respectively.

Figure 7(b) Phase II, New DC-705 Performance Test Data, Effect of Plasticizer Contaminants Added into Ultra Pure DC-705 and Their Removal

With the detail self-cleansing operational philosophy established, the following data Figures 7(b) to (d) will be described in a more comprehensive manner, utilizing data points or significant occurances.

(Event "I") Inserting 4 ml of a phthalate ester mixture containing 2 ml of DIOP, 2 ml DIDP, and 2 ml of DOP/DEHP directly into the highly purified DC-705 diffusion pump boiler after Phase I testing resulted in chamber and foreline pressure increase of 1000 to 10 000 times.

(Event "II") Stabilization of chamber pressure occurred at 7×10^{-4} Torr. Removing 4 ml of L.E. from the top alembric of the foreline obtained the expected improvement in chamber pressure. Chamber pressure was 1.2 to 1.5 decade lower (2.5×10^{-5} Torr) within one hour.

(Event "III") Inserting the remaining 2 ml of phthalate ester contaminant mixture slowly (at a less than 1 ml/min) directly into the boiler duplicated the pressure parameter fluctuation previously. Ten ml of new DC-705 was added so not to deplete the boiler supply during the pending cleansing period. When 2 ml of L.E. fractions were removed twice within a 30 min period, the chamber pressure was reduced three decades within 1 hr, from 3×10^{-3} to 2.5×10^{-6} Torr. The following morning, 15 hr later 8.5×10^{-7} Torr was achieved.

No improvement in chamber pressure occurred until the 1×10^{-6} SPF scale model glass chamber was baked out on the 17th day at 230° F whereupon a 0.25 decade improvement was achieved to 7×10^{-7} Torr. Configuration of the boiler's evaporative surface was 100% working 99% of the time. This behavior pattern was maintained for over three days.

(Event "IV") Lowering the 1×10^{-6} scale test chambers baffle temperature to -10° F in order to study its effect on ultimate chamber pressure were inconclusive. Six days later (26th day of operation) the baffle temperature was reduced to -55° F without an expected corresponding change in chamber pressure. Chamber pressure now was $2-5 \times 10^{-6}$ Torr, up 0.5 decade.

(Event "V") Surmising the existance of light-ends, 1 ml of L.E. was was removed from the top foreline alembric with the immediate increase in pump efficiency indicated by a 0.5 decade decrease in chamber pressure to 7×10^{-7} to 1×10^{-6} Torr. After a 16 hr period of chamber bakeout at 180° F, the lowest ultimate chamber pressure of 5×10^{-7} Torr was observed.

(Event "VI") Inserting 4 ml of phthalate ester mixture containing 2 ml of DIOP, 2 ml DIDP, and 2 ml of DOP/DEHP slowly into the ultra clean test chamber resulted in the expected chamber and foreline pressure increases of 1000 to 10,000 times. Chamber baffle temperature was -55° F.

(Event "VII") Sixteen hours later the chamber pressure was 1.5 to 2×10^{-6} Torr. The bottom of the test chamber revealed condensed plasticizer contaminant. A chamber bakeout at 220° to 280° F was initiated in an attempt to vaporize the newly added plasticizers. An immediate 1.2 decade chamber pressure rise occurred. With the chamber heated to its hottest bakeout temperature (220° to 280° F), two additional milliliters of the phthalate mixture was inserted into the top of the chamber. Chamber pressure increased from the 1×10^{-5} Torr range to 3×10^{-3} Torr range. The chamber baffle was allowed to warm up to 20° F in order to render it ineffective in protecting the highly purified DC-705 in the diffusion pumps. The outgassing load created by the vaporizing plasticizers on the heated chamber walls started to affect the pumps efficiency until chamber pressure stabilized out at $1.5-3 \times 10^{-5}$ Torr. This was a crude simulation of the outgassing from heated (heavily plasticized) cables.

On the 31st day, upon removing 3 ml of L.E. from the top alembric in the foreline, the typical expected improvement process started immediately again. However, due to a power outage in the SPF vacuum technology laboratory sometime that night a discontinuity in the data exists.

(Event "VIII") As the DC-705 pump fluid purity became reinstated, the chamber pressure returned to its prior levels of 6×10^{-7} Torr with a corresponding foreline temperature of 265° F indicating a high molecular throughput. The chamber baffle temperature was -25° F. Warming up the chamber baffle to ambient after the diffusion pump is ultra pure did result in a slight chamber pressure rise of approximately 0.5 decade. Evidently the vast majority of deteriorating contaminants were removed by the self-cleansing process of this diffusion pump vacuum system.

Even though the typical torpid/schizoid/working surface behavior patterns have been repeatedly duplicated with various DC-705 contamination modes, discussion of the photographic/thermovision/movie documentation has been purposely deferred to this section. The test sample was

DC-705 previously exposed to an average gamma dose rate of 25 milli-roentgen (MR)/hr for 5 yr while operating in the vacuum diffusion pump system of the Michigan State University Cyclotron. It had a photographic distinction being discolored by use to a dark brown color. This oil achieved a 2.5×10^{-5} Torr ultimate vacuum in the standard G-4 oil per-formance test, (see Fig. 5). This oil always was handled with the best of vacuum practicies; that is back filling the vacuum system with dry gaseous nitrogen (GN_2) upon diving the cyclotron to atmospheric pres-sure, never thermally shocking it to atmospheric environmental contami-nants, etc.

Figure 7(d) shows after a day of operation that the ultimate test chamber vacuum obtained was 5×10^{-5} Torr. A corresponding chamber baffle temperature of -40° F did not appear to limit the deteriorating effect or reverse fraction of light-end contaminants. For a comparison of boiler evaporative surface behavior patterns as a function of generated ultimate chamber pressure, see Figure 8.

Photograph No. 15, taken at a chamber pressure of 2×10^{-4} Torr, reveals a schizoid pattern of many working holes typical of the silicone pump fluids. A broken light source similar to Hickman's was utilized to high light the evaporative surface.[6] The movie film which is a dynamic documentary portion of this research contains the actual thermovision visual record. The thermovision camera essentially thermally maps what its viewing by utilizing the isotherm markers.[17] The NaCl lense afixed to the boiler allows 90% transmittance between the wave lengths of 0.2 to 13 microns. A typical thermovision polaroid picture of a torpid/schizoid/working evaporative surface with numerous individual working holes is shown next to photograph No. 15.

The other typical thermovision polaroid picture just below reveals a single working hole in a torpid surface. The evaporative surface horizon in the diffusion pump boiler is easily identified. Significant is the local-ized erruption of molecules being released from the working hole, well into the pump boiler vapor cavity. This thermovision photograph con-firms what Hickman described in References 6 and 7, associated with working valleys of different depth. The deepest part of the working val-ley emits the most vapor. Molecular release is greatest from these areas since the evaporative surface is always changing, the size and shape of the working holes are also changing relative to existance, loca-tion, growth, movement, etc.

Two photographs both labeled No. 16 were taken within a few seconds of each other. One taken at a rising chamber pressure of 5 to 10×10^{-5} Torr portrays primarily a torpid surface generating a 0.5 decade higher chamber pressure. The other, a 100% working evaporative surface having an improved chamber pressure of 5×10^{-5} Torr. The evidence of increased molecular flow is everywhere in this photograph; inside the

Christmas tree, at the rim of the individual jets, molecular buildup on the barrel surface, in the foreline, etc.

Marked increase in pump-performance was apparent as soon as light-end and heavy-end contaminants began to be removed from the pump fluid. Within 8 hrs from the start of cleansing-separation-purification processing a 50 times increase in pump performance was achieved as indicated by a 1.5 decade lower chamber pressure varying between 1×10^{-6} to 5×10^{-6} Torr. After the second day of total operation, first full day of contaminant pump fluid cleansing activity, an ultimate chamber pressure varying between 8×10^{-7} to 1×10^{-6} Torr was obtained. The corresponding boiler evaporative surface behavior associated with this pump efficiency was 100% working 90% of the time. The fluid color was still dark brown.

After the first seven(7) days of system operation, the ultimate chamber vacuum fluctuated between 5×10^{-7} and 1×10^{-6} Torr with a corresponding improved evaporative surface now being 100% working 99% of the time. All other critical operating parameters were similar to those of the previous tests. Points "A" and "B" confirmed the interdependancy between all critical parameters and minute changes in chamber baffle temperature. This is evident when even at nominal baffle temperatures of -40 to -15° F. Significant were the occasional higher isolated pressure rise spikes caused by the total 100% working surface startups, as noted in points "B_1" and "B_2".

Point "C" shows again that further increase in pump efficiency (decrease in chamber pressure) can be expected as long as there are some light-ends available for removal, regardless of the chamber baffle temperature fluctuations.

Point "D" indicates achievement of a continuous 100% working evaporative surface. Point "E" shows the effect of changing the air cooling flow/distribution in the SPF Vacuum Technology Laboratory and/or the change increase in diffusion pump heater voltage.

Point "F" is noteworthy in that the color of the boiler fluid changed from dark brown to light yellow overnight after the heater voltage was increased approx. one volt. Expected effect of putting additional ion gages on is also shown. See photographs No. 19 for further evidence of increase in molecular flow throughout the entire pump when evaporative surface is continuously 100% working.

An investigation into the effectiveness of the chamber baffle at 5×10^{-7} Torr vacuum range was undertaken. The effect of the first major baffle temperature warmup cycle to 40° F on all critical parameters are indicated by event "I". Respective baffle temperature warmup cycles are revealed by events "II", "III", "IV", "V", and "VI". As Huntress[9] implied, and the foregoing results confirm, if fluid vapor pressure is maintained at its lowest absolute value (10^{-10} Torr for DC-705) corresponding to an ultra high purity condition, many hours of vapor-free testing are provided with no more than a watercooled baffle. This could pro-

vide a significant operational cost reduction to large space simulation facilities.

Photograph No. 21 reveals the violent-dynamics of a continuous 100% working evaporative surface. Note, the partially bare boiler bottom, the vapor recoil action, the gobs of liquid actually carried along with the high molecular release rate from the boiler fluid, and the heavy buildup of molecules/liquid formations throughout all portions of the diffusion pump assembly.

Events "VII" and "VIII" reveal the criticality of the proper amount of boiler heater input in order to successfully maintain a continuous 100% working evaporative surface with its associated increase in optimum diffusion pump efficiency.

SUMMARY OF RESULTS AND CONCLUDING REMARKS

1. Torpidity, a menace to high vacuum pot stills but not considered at present a serious factor in the vapor pumps, is a definite factor in reducing the pumps efficiency by factors of 100 times. The scale model (self-cleansing) glass diffusion pump data showed 100% working evaporative surfaces produce ultimate pressures 100 to 1000 times lower than those of torpid evaporative surfaces. It is surmised that torpidity is the result of an increased surface tension phenomena caused by impurities collecting on the evaporative surface, acting as a barrier limiting the molecular release rate.

2. Where no specific chemical has previously been known which will confer torpidity when added to a nontorpid liquid, 1×10^{-6} SPF-scale model test data showed that phthalate esters, namely DIOP and/or DIDP, added directly into the boiler fluid or vacuum system chamber caused a nontorpid surface to become partially schizoid (100% working 50% of the time) with a corresponding increase in pressure of three decades. Analyses of light-end and heavy-end fractions separated from contaminated (torpid/schizoid producing) DC-705 silicone oil detected phthalate ester plasticizers.

3. Where further increases in specific pumping speed have not been considered likely, the scale model (self-cleansing) glass diffusion pump, incorporating contamination removal technology innovations, consistently produced ultimate pressures that were for new DC-705 oil, 1.5 decade lower; for 5 yr old DC-705, over 2 decades lower; and for new DC-705 oil intentionally contaminated with phthalate esters, nearly 3 decades lower. Diffusion pump innovations were successfully tested allowing removal of contaminants from pump oil during operation. Vacuum distillation removal of light-end fraction contaminants and boiler bleed removal of heavy-ends from the evaporative surface resulted in a 2 decade or more lower ultimate pressure.

4. When self-purification of the intentionally contaminated diffusion pump oil was not allowed, no significant improvement in ultimate vacuum

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was achieved after continuous operation in excess of one month, thereby indicating the necessity of removing these phthalate esters.

5. The color of DC-705 is not indicative of purity or capability for obtaining maximum molecular release conditions from an evaporative surface, since similar ultimate vacuum levels and conditions were realized with a dark brown color oil as with a lighter color oil.

6. A water baffle at ambient temperature may be all that is required for a high efficiency diffusion pump, provided the boiler fluid is maintained at an ultra high purity condition (fluid vapor pressure kept at its lowest absolute value - 10^{-10} Torr for DC-705) by vacuum systems incorporating contaminate removal technology innovations.

7. Contaminants, commonly found in large vacuum chambers, that adversely affected silicone oil diffusion pump performance were identified as phthalate ester plasticizers. These plasticizers are utilized in cable insulation jackets, alkyd paints, electronic components, etc. Those having the most deteriorating effect on DC-705 being: DIOP 390 M.W., DTDP 530 M.W., DIDP 446 M.W., and DOP/DEHP 390 M.W., respectively.

8. Contaminated DC-705 diffusion pump oil can be successfully reprocessed by high vacuum molecular distillation with an approx. 75% yield and capable of achieving an ultimate vacuum close to that of new DC-705 oil.

9. Further study to improve the purification-separation concepts described, methods of measuring changes in molecular release rates, and spectroscopy fingerprints identifying contaminating constituents and related physical properties on the evaporative surface is warranted.

ACKNOWLEDGMENTS

REFERENCES

1. Fenn, David B.; Deyo, James N.; Miller, Thomas J.; and Vernon, Richard W.: Experimental Performance of a 2-15 Kilowatt Brayton Power System in the Space Power Facility Using Krypton. NASA TM X-52750, 1970.
2. Daye, Charles J.: Skylab Payload Shroud Jettison Tests. NASA TN D-6913, 1972.
3. Buggele, Alvin E.; and Lohwater, Robert K.: Improved Method for Reclaiming Vacuum Diffusion Pump Oil. NASA Tech Brief B72-10511, August 1972.

4. Burrows, G.: Molecular Distillation. Oxford University Press, Amen House, (London) 1960.

5. Watt, Peter R.: Molecular Stills. Reinhold Publishing Corp., 1963.

6. Hickman, Kenneth: Torpid Phenomena and Pump Oils. Jour. of Vacuum Science and Technology, Vol. 9, No. 2, Jan./Feb. 1972, pp. 960-976.

7. Hickman, K.; Maa, J. R.; Harris, P. J.; and Davidhazy, A.: The Effects of Surface Properties of Water on Evaporation and Condensation. R&D Progress Rept. No. 585, Rochester Institute of Technology, for Office of Saline Water, U.S. Dept. of the Interior, Grant No. 14-01-0001-2119, Dec. 1970.

8. Huntress, Arnold R.: Clean Diffusion Pump Fluids, How They Stay That Way. Dow Corning Corp. (Presented Amer. Vac. Soc. Symposium - Abstract 1966, unpublished).

9. Huntress, Arnold R., and Schmock, Walter R.: A Direct Analytical Method for Measuring the Condition of Diffusion Pump Fluids. Dow Corning Corp. (Presented Amer. Vac. Soc. Symposium - Abstract 1965, unpublished).

10. Hablanian, M. H.; and Maliakal, J. C.: Advances in Diffusion Pump Technology, Jour. of Vacuum Science and Technology, Vol. 10, No. 1, Jan./Feb. 1973, pp. 58-64.

11. Krell, Erich; and Lumb, E. C.: Handbook of Laboratory Distillation, Elsevier Publishing Co., 1963.

12. Perry, E. S.; and Weissberger, A.: Distillation: Technique of Organic Chemistry. Vol. IV, Interscience Publishers, 1965.

13. Fisher, Aaron; and Mermelstein, Benjamin: GSFC Micro-volatile Condensable Materials System for Polymer Outgassing Studies. NASA GSFC X-735-69-471, 1969.

14. Gross, Frederick C.; and Colony, Joe A.: An Etiological Study of Phthalate Self-Contamination of Spacecraft and Contamination from Their Earthly Environs. NASA TN D-6903, 1972.

15. Winspear, George G. (editor): The Vanderbilt Rubber Handbook. R. T. Vanderbilt Co., Inc., 1968.

16. Infrared Spectroscopy Committe, Chicago Society for Paint Technology: Infrared Spectroscopy - It's Use in the Coating Industry. Federation of Societies for Paint Technology, 1969.

17. Borg, Sven. Bertil.; Thermal Imaging with Real Time Picture Presentation. Applied Optics, Vol. 7, Sept. 1968, pp. 1697-1703.

TABLE I. - SOME SPF TEST SUPPORT MATERIALS ASSOCIATED WITH DC-705 OIL CONTAMINATION PROBLEM

(a) Volatile (Outgassing) Materials and Their Analyses

Material description/type	Manufacturer	Total quantity exposed, ft	Length of time exposed to vacuum, hrs	GSFC outgassing test results[c]		KSC DTA test results 25°-340° C, %	General information provided by manufacturer or by KSC analyses
				Wt. loss, %	VCM, %	25°-360° C, to 380° C, %	
Cable							
Sample (5a) #9404 3C or 4C shielded cable	Belden	18 000	0	----	---	63.5	Approximately 50% PVC resin, 25% DIDP
Sample (5a) #9404 3C or 4C shielded cable	Belden	18 000	3000	13.9	9.5	58	Approximately 50% PVC resin, 25% DIDP
Sample (5b) #8262 RG-58C/U shielded coax	Belden consolidated	1 000	0	----	---	31	Nitrile modified PVC (no phthalate ester plasticizers used) in Geon #720
Sample (5b) #8262 RG-58C/U shielded coax	Belden Consolidated	1 000	3000	2.9	1.2	30	Nitrile modified PVC (no phthalate ester plasticizers used) in Geon #720
Neoprene	Dupont			3.01-7.95	1.5-6.14	33.6 (25°-300° C)	
#16 Awg. 3C SO cable	Carol Cable Co.	2 500	3000	----	---	----	
3/4 in. B-ring seal	Irving B. Moore	490	3000	----	---	----	Neoprene type rubber with high aromatic amine antioxidant level, inner layer— a butyl rubber (polyisobutylene)
350 MCM cable (samples d)	Paramite Cable	290	3000	----	---	37	
Samples #17, #18, and #21	Carol, Apex, and	2500, 2000,	40, 3065,	----	---	----	PVC resin with phthalate ester plasticizer and carbonate and silica filler for samples #17, #18, and #21 outerjacket only. #21 Mylar type
#19 Awg., #12 Awg., and #18 Awg.,	Belden or	and 10 000	and 60				
single conductor or shielded cable	Ray Chem	500 ft²					Terephthalate ester wrap with polyethylene conductor insulation
Polar crane							Gray alkyd oil paint typical
Paint samples							
2(a) green[d]	P & H Hoist	65	6.5[d]	2.0[d]		----	Phthalic anhydride, M.W. 148.1 (approx. 24%), alkyd binder, titanium oxide and traces of DC-705
2(b) red primer under gray[e]	Rustoleum & Sherman Williams	65	3.8[e]	0.4[e]		----	Phthalate type (butyl phthalate, M.W. 278), alkyd binder, talc, silica
3(c) gray paint[f]	General Electric	65	6.7[f]	2.0[f]		----	A nonphthalate ester alkyd, carbonate, sulfate, titanium oxide, traces of DC-705
3(a) green[d]	P & H Traction Drive Motor	65	6.1[d]	1.5[d]		----	Ratio of 665 hr to 65 hr outgassing rates is 88%, ratio of 665 hr to 65 hr VCM rate is 79%
4(a) light gray[e]	G. E. control cable door	3065	3.8[e]	0.8[e]		----	Ratio of 3065 hr to 65 hr outgassing rates is 95%, ratio of 3065 hr to 65 hr VCM rate is over 100% due to silicone oil being absorbed with time
4(b) dark gray[f]	G. E. control cable door	3065	4.9[f]	1.1[f]		----	Ratio of 3065 hr to 65 hr outgassing rates is 73%, ratio of 3065 hr to 65 hr VCM rate is 56%
4(c) black	Mercury vapor light	3065	1.7	0.4		----	No data given
Sample #35 Catch Net, structure paint	Unknown	3 000 ft²	----	---		----	Phthalate type alkyd binder, organic salt (rosin salt, probably zinc oxide) and traces of DC-705 oil

(b) Contaminated - Reprocessed Oil Analyses[b]

Sample No.	DC-705 (Dow Corning) Oil Sample Description	Color	Analyses using infrared spectrophotometric techniques at KSC
41	3046 hr old, 10% precut distillation of light-end contaminants	clear white	Detected a phthalate-type ester or alkyd
42	3060 hr old, 90% final cut distillation of heavy-ends contaminants	dark brown	Detects a phthalate-type ester alkyd, a polyurethane resin
44	3040 hr old SPF DC-705	brown	Some phthalate ester detected, carbonaceous material showing carbonyl character and silicone character, probably a degradation of reaction product

(c) 1×10⁻⁶ Scale Model Contaminated Oil Analyses[b]

II 3-7-73 Contaminated DC-705 oil samples (taken post-insertion of 6 ml mixture of phthalate plasticizers - 2 ml DIDP, 2 ml DIOP, 2 ml DOP DEHP directly into boiler (bulk)(color, clear white)
  Sample #5 - Light-end fractions from foreline; color, pure white; 18.4% phthalate ester content

VIII 3-29-73 Contaminated DC-705 oil samples (taken post-insertion of 6 ml mixture of phthalate plasticizers - 2 ml DIDP, 2 ml DIOP & 2 ml DOP DEHP directly into top of chamber)(color, clear white)
  Sample #4 - Post test residual boiler fluid - color, pure white; 0.7% phthalate ester content[a]
  Sample #5 - Post test light end fractions from foreline; color, pure white; 8% phthalate ester content

VIII 4-19-73 Post test Michigan State cyclotron 5 yr old DC-705 oil samples (color, dark brown):
  Sample #1 - Light end fractions from foreline; color, clear white; 1.2% phthalate ester content
  Sample #2 - Skimmed heavy ends from evaporative surface; color, deep yellow, 0.7% phthalate ester content[a]

[a]One percent is the least quantity detectable with any certainty, due to instrument system noise and or no assurance that curve goes to zero.
[b]A FTS-20 infrared fourier transform spectrometer was used to determine the phthalate ester content of the DC-705 silicone oil samples.
[c]521 Perkin Palmer spectrophotometer.
[d]Identical sources.
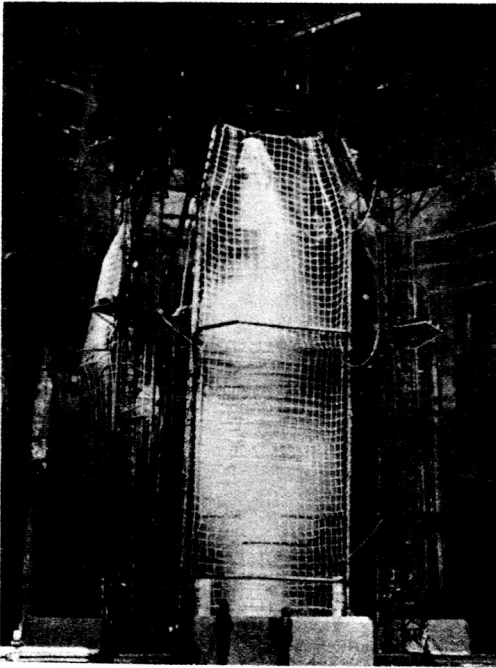[e]Identical sources.
[f]Identical sources.
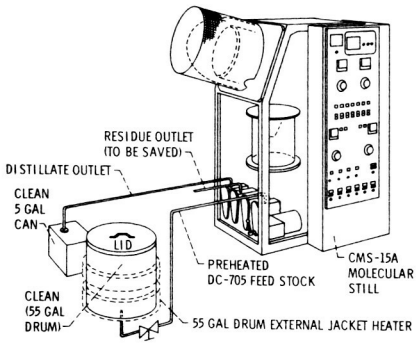
Figure 1. - Skylab payload shroud jettison test setup in SPF chamber.
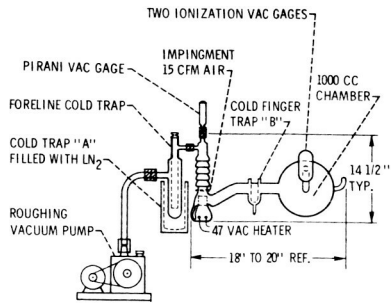

Figure 2. - Typical DC-705 reprocessing setup.

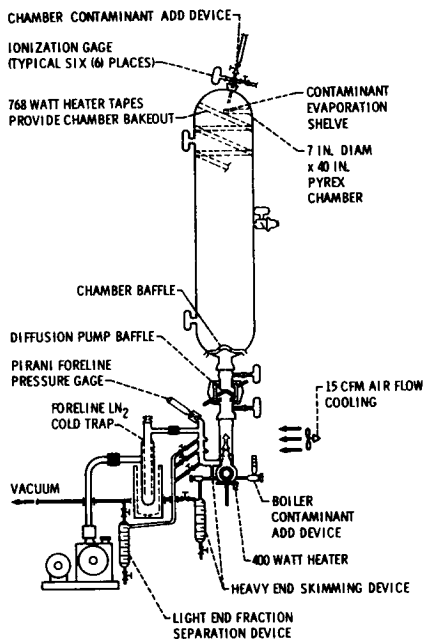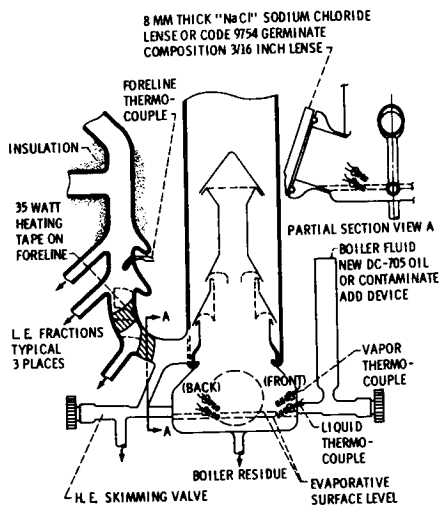Figure 3. - Typical DC-705 oil performance evaluation setup.

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CHAMBER CONTAMINANT ADD DEVICE

IONIZATION GAGE
(TYPICAL SIX (6) PLACES)

768 WATT HEATER TAPES
PROVIDE CHAMBER BAKEOUT

CONTAMINANT
EVAPORATION
SHELVE

7 IN. DIAM
x 40 IN.
PYREX
CHAMBER

CHAMBER BAFFLE

DIFFUSION PUMP BAFFLE

PIRANI FORELINE
PRESSURE GAGE

FORELINE LN$_2$
COLD TRAP

15 CFM AIR FLOW
COOLING

VACUUM

BOILER
CONTAMINANT
ADD DEVICE

400 WATT HEATER

HEAVY END SKIMMING DEVICE

LIGHT END FRACTION
SEPARATION DEVICE

(a) 1x10^{-6} SPF SCALE MODEL GLASS 2ND GENERATION VACUUM SYSTEM
INCORPORATING CONTAMINATION REMOVAL TECHNOLOGY.

8 MM THICK "NaCl" SODIUM CHLORIDE
LENSE OR CODE 9754 GERMINATE
COMPOSITION 3/16 INCH LENSE

FORELINE
THERMO-
COUPLE

INSULATION

35 WATT
HEATING
TAPE ON
FORELINE

PARTIAL SECTION VIEW A

BOILER FLUID
NEW DC-705 OIL
OR CONTAMINATE
ADD DEVICE

L. E. FRACTIONS
TYPICAL
3 PLACES

VAPOR
THERMO-
COUPLE

(BACK)   (FRONT)

LIQUID
THERMO-
COUPLE

BOILER RESIDUE

EVAPORATIVE
SURFACE LEVEL

H. E. SKIMMING VALVE

(b) EXPOSED VIEW OF 1x10^{-6} SPF SCALE MODEL GLASS 2ND
GENERATION DIFFUSION PUMP INCORPORATING CON-
TAMINATION REMOVAL TECHNOLOGY.

NOTE: TYPICAL ALEMBRIC GUTTERS
WITH OR WITHOUT STAINLESS STEEL
FRACTIONATING COLUMN PACKING
0.090" x 0.175" SIZE C, PODBIELNIAK
HELI-PAK NO. 3008 COLUMN PACKING

INSULATION

INSULATION

L. E. FRACTIONS
TYPICAL 4 PLACES

35 WATT HEATING
TAPE WRAPPED
ABOUT FORELINE

MINIATURE STAINLESS
STEEL SHEATHED
MAGNESIUM OXIDE
COPPER CONSTANTAN
THERMOCOUPLE 4 TYP.

H. E. SKIMMING VALVE

HIGH THROUGHPUT CONFIGURATION

HIGH SEPARATION - PURIFICATION CONFIGURATION

(c) PARTIAL EXPOSED VIEW OF 1x10^{-6} SPF-SCALE MODEL GLASS
2ND GENERATED DIFFUSION PUMP INCORPORATING CON-
TAMINATION REMOVAL TECHNOLOGY.

Figure 4

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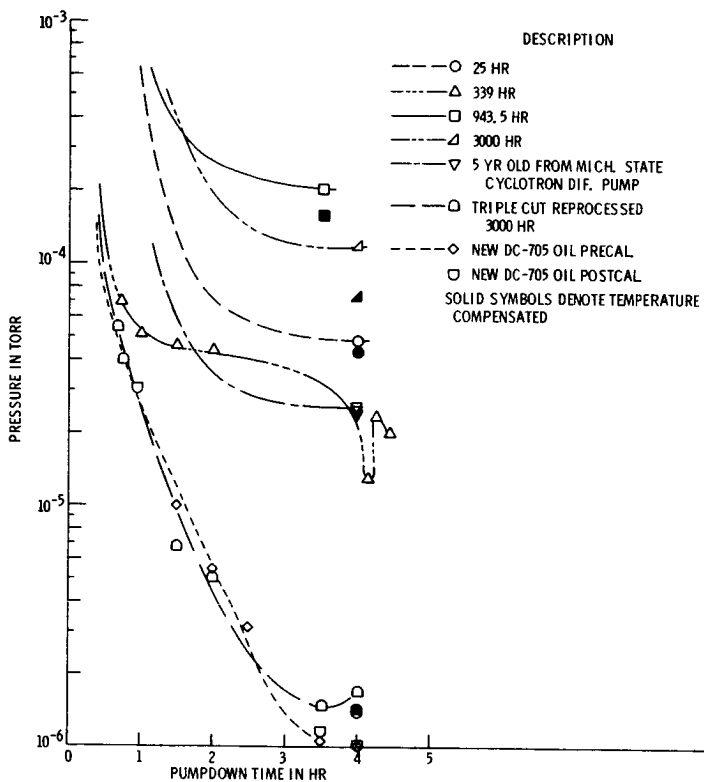
Figure 5. – Performance of DC-705 oil compared with new and reprocessed oil utilizing a (G-4) single stage glass diffusion pump.
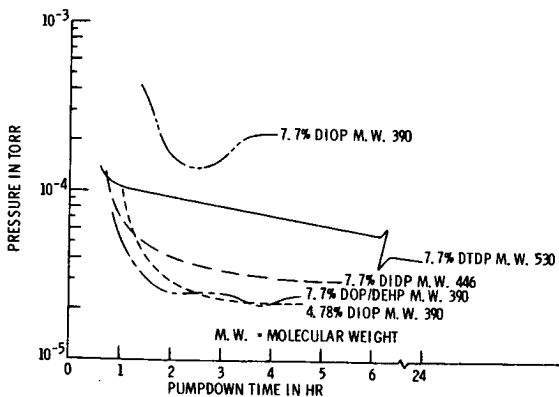

Figure 6. – Diffusion pump performance of DC-705 oil with various contaminants utilizing a (G-4) single stage glass diffusion pump.
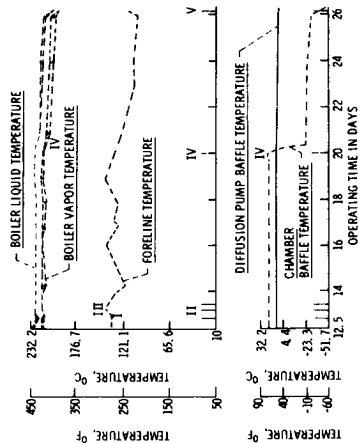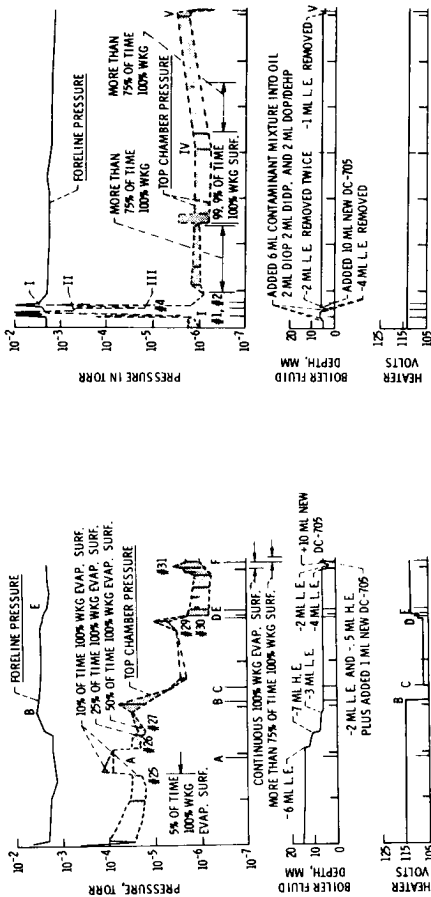
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FORELINE PRESSURE

MORE THAN 75% OF TIME 100% WKG

MORE THAN 75% OF TIME 100% WKG

TOP CHAMBER PRESSURE

99.% OF TIME 100% WKG SURF.

ADDED 6 ML CONTAMINANT MIXTURE INTO OIL 2 ML DIOP 2 ML DIDP, AND 2 ML DOP/DEHP
-2 ML L E REMOVED TWICE   -1 ML L E REMOVED
ADDED 10 ML NEW DC-705
-4 ML L E REMOVED

BOILER LIQUID TEMPERATURE

BOILER VAPOR TEMPERATURE

FORELINE TEMPERATURE

DIFFUSION PUMP BAFFLE TEMPERATURE

CHAMBER BAFFLE TEMPERATURE

OPERATING TIME IN DAYS

PRESSURE IN TORR

BOILER FLUID DEPTH, MM

HEATER VOLTS

TEMPERATURE, °C

TEMPERATURE, °F

Figure 7(b). - Phase II, new DC-705 performance test data, plasticizer contaminants added into ultra pure DC-705 and their removal.

FORELINE PRESSURE

10% OF TIME 100% WKG EVAP. SURF.
25% OF TIME 100% WKG EVAP. SURF.
50% OF TIME 100% WKG EVAP. SURF.

TOP CHAMBER PRESSURE

CONTINUOUS 100% WKG EVAP. SURF.
MORE THAN 75% OF TIME 100% WKG SURF.

5% OF TIME 100% WKG EVAP. SURF.

-6 ML L E.

-7 ML H.E.   -2 ML L.E.
-3 ML L.E.   -4 ML L.E.   +10 ML NEW DC-705

-7 ML L.E. AND - 5 ML H.E.
PLUS ADDED 1 ML NEW DC-705

-2 ML L.E.

BOILER LIQUID TEMPERATURE

BOILER VAPOR TEMPERATURE

FORELINE TEMPERATURE

DIFFUSION PUMP BAFFLE TEMPERATURE

CHAMBER BAFFLE TEMPERATURE

OPERATING TIME IN DAYS

PRESSURE, TORR

BOILER FLUID DEPTH, MM

HEATER VOLTS

TEMPERATURE, °C

TEMPERATURE, °F

Figure 7(a). - Phase I, new DC-705 performance test data, increased purification of new DC-705 nets 1.5 decade.
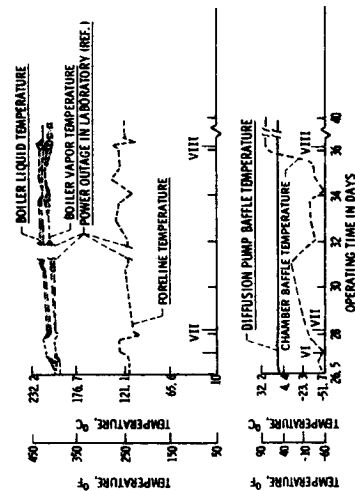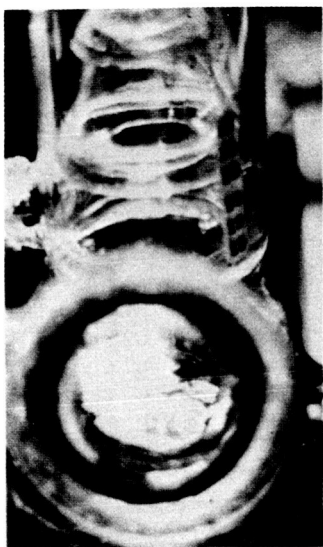
304
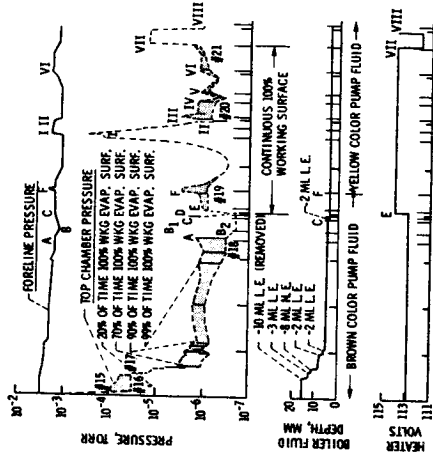
Figure 7(b). - 5 year old Michigan State cyclotron DC-705 oil performance test data with the 1x10⁻³ SPF scale model self-cleansing pump.
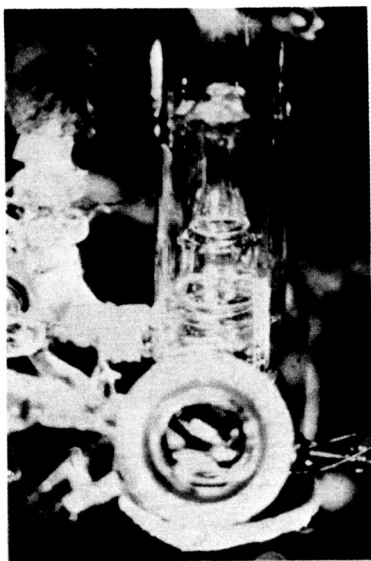
Figure 7(c). - Phase III, new DC-705 performance test data, plasticizer contaminants added into chamber and their removal with cold and warm baffles
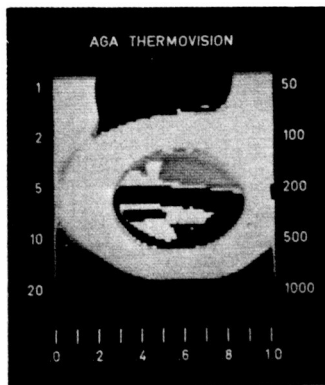
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TORPID/SCHIZOID WORKING EVAPORATIVE
SURFACE, VACUUM 2×10^{-4} TORR AT TOP
OF CHAMBER. (PHOTOGRAPH # 15).
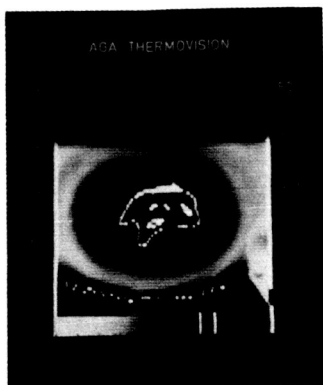
TYPICAL THERMOVISION MONITOR POLA-
ROID PICTURE OF A TORDID/SCHIZOID/
WORKING EVAPORATIVE SURFACE.

TORPID/SCHIZOID WORKING EVAPORATIVE
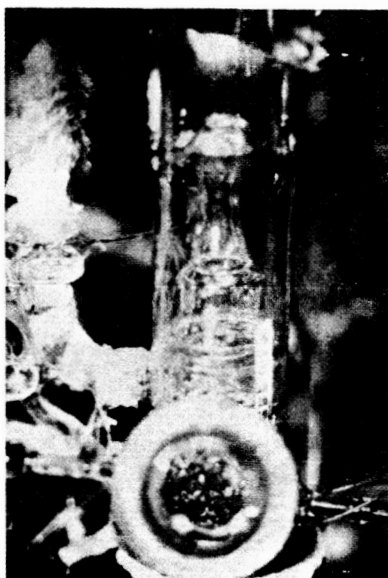SURFACE, VACUUM 10×10^{-5} TORR AT TOP OF
CHAMBER. (PHOTOGRAPH # 16).

TYPICAL THERMOVISION MONITOR POLA-
ROID PICTURE OF VAPOR BEING RELEASED
FROM A WORKING HOLE.

Figure 8. - Typical corresponding slide, thermovision/polaroid picture of a torpid/schizoid/working
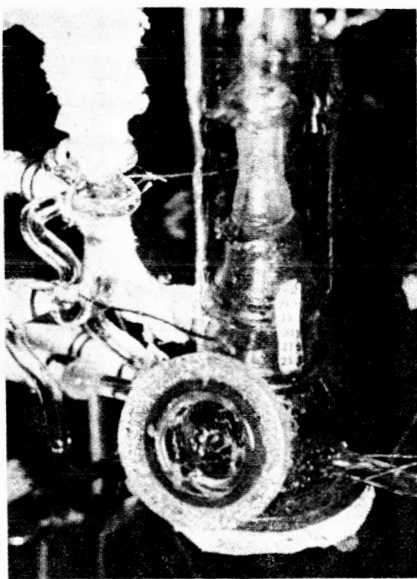surface.

306
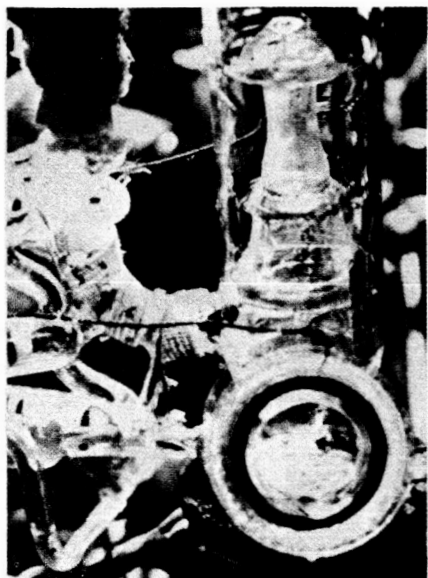
100 PERCENT WORKING EVAPORATIVE SURFACE (CONTINUOUS), VACUUM AT TOP OF CHAMBER $1.5-4 \times 10^{-6}$ TORR, BAFFLE TEMPERATURE 70° F AT PUMP AND -25° F AT CHAMBER. (PHOTOGRAPH #19)

100 PERCENT WORKING EVAPORATIVE SURFACE (APPROXIMATELY 20 PERCENT OF TIME), VACUUM AT TOP OF CHAMBER 5×10^{-5} TORR. (PHOTOGRAPH #16)
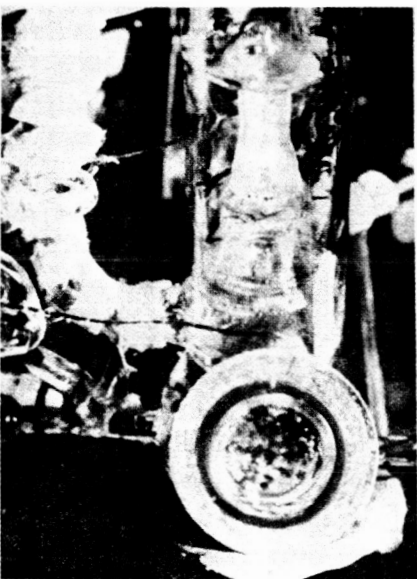
100 PERCENT WORKING EVAPORATIVE SURFACE (CONTINUOUS) SAME CONDITIONS AS ABOVE BUT SURFACE APPEARS MORE DYNAMIC. (PHOTOGRAPH #19)

100 PERCENT CONTINUOUS WORKING EVAPORATIVE SURFACE, VACUUM AT TOP OF CHAMBER 1×10^{6} TORR, 20 TO 30 PERCENT OF BOILER BOTTOM APPEAR BARE DUE TO VAPOR RECOIL ACTION. (PHOTOGRAPH 21)

Figure 8. - Concluded.